Name of Procedure:

Infrared Spectrophotometry

Suggested Uses:

Collection of qualitative data for the identification of controlled and non-controlled substances.

Apparatus Used to Perform Procedure:

Perkin-Elmer models: Spectrum One and Spectrum 100
Perkin-Elmer Universal Attenuated Total Reflectance (ATR) Sampling Accessory
Printer/ink cartridge
Paper
Spatula
Polystyrene film standard
Polystyrene film standard spectra (attachment)

If the KBr method is used:
Potassium bromide (infrared grade)
Wig-L-Bug grinding mill
Stainless steel vial and ball
Hydraulic press
Mortar and pestle
Potassium bromide salt plates and holder
Pellet holder
KBr pellet die (13mm)
Oven
Thermometer

Performance Verification Requirements:

Each IR instrument shall have a monthly performance verification to ensure proper functioning.

The internal polystyrene of each IR instrument shall be compared to a traceable standard yearly.

Performance Verification Procedure:

Monthly:

Instructions for Spectrum One/Spectrum 100 Performance Verification:

1. Double click on the "Spectrum Software" icon
   Login – click OK through prompts
2. Scan Background
   Click on "Scan and Instrument Setup" hotkey
   ("Scan" tab – check for Scan duration set to 4)
   ("Instrument" tab – check for Resolution set to 4.00 cm^{-1})
   "Background" hotkey
   Scan
3. Scan using internal polystyrene
   Click on "Scan and Instrument Setup" hotkey
   "Beam" tab
   Double click on picture with disk/holes on the left
   Choose "Polystyrene"
   OK
   Apply
   Scan
4. Click "Label Peaks" hot key
   Print the scan with at least the following three wave numbers marked by the Label Peaks software:

   3082.25 cm^{-1} (+/- 0.5 cm^{-1})
   1601.33 cm^{-1} (+/- 0.5 cm^{-1})
   1028.47 cm^{-1} (+/- 0.5 cm^{-1})

5. Reset the beam and instrument for casework samples:
   Click on "Scan and Instrument Setup" hotkey
   "Beam" tab
   Double click on picture with disk/holes on the left
   Choose "None"
   OK (This removes the polystyrene from the beam.)

Apply
"Contamination check" hotkey
OK (This runs a contamination check in preparation for samples.)

6. The allowable variance from the certified value for the peaks appearing at these wave numbers is +/- 0.5 cm^{-1}. If the results are outside these specifications, the instrument will be removed from casework immediately and the following shall be done:

   a. Place an "Out of Service" sign on the front of the instrument.
   b. Notify the IR Coordinator so he/she can call the Service Engineer to schedule an on-site assessment.

7. The monthly polystyrene scan will be filed and maintained by the IR Coordinator in a notebook that is kept with each instrument.

Yearly:
Internal Polystyrene Verification:

A scan of a certified, traceable polystyrene film shall be collected with the KBr accessory in place, followed by the collection of a scan of the internal polystyrene with the ATR attachment in place, according to the procedure above. The data obtained shall be evaluated using the criteria in the procedure above. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

New Instrument Set Up:

1. New FTIR instruments will be installed by a Perkin-Elmer engineer according to Perkin-Elmer policy.
2. Upon completion of the installation a scan of a certified, traceable polystyrene film shall be collected with the KBr accessory in place, followed by the collection of a scan of the internal polystyrene with the ATR attachment in place, according to the procedure above. The data obtained shall be evaluated using the criteria in the procedure above. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

3. Scans from at least three controlled substances will also be obtained, following polystyrene confirmation, at instrument setup. Examples include: Methamphetamine, Phentermine, and Cocaine Base, but other controlled substances may be used depending on availability of standards. The data obtained shall be reviewed by the IR Coordinator and found to be substantially the same as the library standard for that compound. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

Application of Procedure for Solid Samples Using the ATR:

1. Clean the attenuated total reflectance (ATR) sampling accessory crystal using water or an organic solvent. Ensure that the crystal is completely dry.
2. Perform a contamination check to ensure that the crystal has been properly cleaned. If the contamination check fails, repeat the cleaning procedure.
3. Place approximately 1 milligram of sample evenly onto the ATR crystal.
4. Apply appropriate force using the ATR force arm to ensure good contact between the sample and the surface of the crystal.
5. Scan to acquire data.
6. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
7. Print the completed scan and compare it to a known reference standard.

Application of Procedure for Liquid Samples Using the ATR:

1. Clean the attenuated total reflectance (ATR) sampling accessory crystal using water or an organic solvent. Ensure that the crystal is completely dry.
2. Perform a contamination check to ensure that the crystal has been properly cleaned. If the contamination check fails, repeat the cleaning procedure.
3. Apply enough of the liquid sample to cover the ATR crystal.
4. Scan to acquire data.
5. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
6. Print the completed scan and compare it to a known reference standard.

Application of Procedure for Solid Samples Using the KBr Method:

1. Place approximately 1 milligram of sample and approximately 100 milligrams of

potassium bromide in the capsule.
2. Grind the mixture in Wig-L-Bug and transfer to the pellet die assembly.
3. Press the pellet die assembly (vacuum optional) in the pellet press and remove the pellet.
4. Place the pellet in the pellet holder.
5. Check background and obtain new background if needed. (Scan empty sample area to acquire data into background region.)
6. Place sample pellet holder in sample area of instrument and scan to acquire data.
7. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
8. Print the completed scan and compare it to a known reference standard.

Application of Procedure for Liquid Samples Using the KBr Method:

1. Place approximately 100 milligrams of potassium bromide in the capsule.
2. Grind in the Wig-L-Bug and transfer to the pellet die assembly.
3. Press the pellet die assembly (vacuum optional) in the pellet press and remove the pellet.
4. Place the pellet in the pellet holder.
5. Smear a thin layer of the liquid sample on one side of the pellet.
6. Check the background; obtain a new background if needed.
   (Scan the empty sample area to acquire data into background region.)
7. Place the sample pellet holder in the sample area and scan to acquire data.
8. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
9. Print the completed scan and compare it to a known reference standard.

Application of Procedure for Volatile Liquid Samples Using the KBr Method:

1. Check the background; obtain a new background if needed. (Scan empty sample area to acquire data into background region.)
2. Place a sample of the liquid into a shallow, open container.
3. Place this immediately inside the sample area of the instrument and close the cover.
4. Wait for approximately one minute or until the sample area is saturated with the sample vapors.
5. Scan to acquire data.
6. Data can now be processed in any number of ways including: flattened, abexed and rescaled.

7. Print the completed scan and compare it to a known reference standard.

Safety Concerns:

ATR Method
Make sure powder does not come into contact with eyes.
Do not over tighten the force gauge.
Be careful with organic solvents so they do not come into contact with eyes.

KBr Method
Make sure capsule is firmly seated in Wig-L-Bug before operating apparatus.
Do not exceed 10 tons of pressure in the pellet press.
Do not look directly into the laser beam emitted from the infrared spectrophotometer.

Literature References:

Moffat, A. C. Ed., **Clarke's Isolation and Identification of Drugs**, 2nd. Ed., The Pharmaceutical Press, 1986.

Mills, III, Terry and Roberson, Conrad J., **Instrumental Data for Drug Analysis**, 2nd Ed., Vols. 1-5, CRC Press, Inc., 1993.

Silverstein, R. M. And Brassler, Clayton G., and Terence C. Morrill, **Spectrometric Identification of Organic Compounds**, New York, Wiley, 1991.

Keller, Roger, **The Sigma Library of FT-IR Spectra**, Edition 1, Vol. 1 and 2, Sigma Chemical Company, Inc., 1986.

Pouchert, Charles J., **The Aldrich Library of Infrared Spectra**, Aldrich Chemical Company, 1981.